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IS 9850 (2004): Methyl iso-Butyl Ketone [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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मिथाईल आइसो-ब्यूटाईल कीटोन — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

METHYL iso-BUTYL KETONE — SPECIFICATION

(*First Revision*)

ICS 71.080.80

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

March 2001

Price Group 2

Alcohols and Allied Products Sectional Committee, PCD 10

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1981. The committee responsible for the formation of this standard decided to revise the same in the light of the experience gained over the years and to align the same with the latest development in technology. Requirements for colour, water content, acidity and alcoholic impurities have been modified. Requirement for purity has been included. The test procedure for the determination of methyl iso-butyl carbinol (alcoholic impurity) has been modified.

Methyl iso-butyl ketone is a colourless liquid with non-residual odour. Its solubility in water is low, but it is completely miscible with a number of organic liquids which makes it an excellent solvent for a wide variety of natural and synthetic resins, lacquers and antibiotics. It is also used in the de-waxing of lubricating oils as well as in the separation of lubricating oil fractions in the lube refineries. It is also employed in the manufacture of flavouring materials, perfumes, pharmaceuticals, dyestuffs, synthetic resins and rubber accelerators.

Considerable assistance has been drawn from ASTM D 1153:1994 Specification for methyl iso-butyl ketone.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHYL iso-BUTYL KETONE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for methyl iso-butyl ketone (4-methyl-pentane-2-one).

2 NORMATIVE REFERENCES

2.1 The following standards contain provisions, which through reference in this text constitute the provisions of the standards. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below:

IS No.	*Title*
229 : 1993	Ethyl acetate — Specification (*third revision*)
878 : 1975	Graduated measuring cylinders (*first revision*)
1070 : 1992	Reagent grade water (*third revision*)
1260 (Part 1) : 1973	Pictorial markings for handling and labeling of goods: Part 1 Dangerous goods (*first revision*)
2362 : 1973	Determination of water by Karl Fischer method (*first revision*)
5298 : 1983	Method for determination of distillation range and distillation yield (*first revision*)
8768 : 1988	Method of measurement of colour in liquid chemical products in Hazen units (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be clear and free from sediments and also shall be free from matter in suspension and shall consist essentially of methyl iso-butyl ketone $[CH_3COCH_2CH(CH_3)_2]$.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A to F. Reference to the relevant clauses of Annex is given in col 4 of Table 1.

Table 1 Requirements for Methyl iso-Butyl Ketone
(*Clause* 3.2)

Sl No.	Characteristic	Requirements	Method of Test, Ref to	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Colour, Pt-Co Scale Hazen units, *Max*	15	—	8768
ii)	Relative density, @ 27/27 °C	0.794 to 0.799	A	—
iii)	Distillation range:			
	a) Initial boiling point @ 760 mm Hg, *Min*	114 °C	B	—
	b) Dry point @ 760 mm Hg, *Max*	117 °C		
iv)	Water content, percent by mass, *Max*	0.1	C	—
v)	Acidity (as acetic acid), percent by mass, *Max*	0.01	D	—
vi)	Non-volatile matter, mg/100 ml, *Max*	5	E	—
vii)	Methyl iso-butyl ketone, percent by mass, *Min*	99.0	F	—
viii)	Methyl iso-butyl carbinol, percent by mass, *Max*	0.3	F	—

4 PRECAUTION IN HANDLING

4.1 Methyl iso-butyl ketone is considered as practically non-toxic but splashes in the eye cause irritation and can result in injury. Eye shields should be worn during handling operations. Splashes in the eye should be removed immediately by washing with copious quantities of water.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in sound and clean containers as agreed to between the purchaser and the supplier. The container shall be stored in a place protected from direct sunlight.

5.2 Marking

The container shall be suitably marked with the following information:

a) Name of the material;

b) Net mass of the material in the container;
c) Name of the manufacturer and his recognized trade-mark if any;
d) Batch number or lot number, in code or otherwise; and
e) Date of manufacture.

5.2.1 Each container shall have the caution label 'FLAMMABLE' together with the corresponding symbol for labeling of dangerous goods [*see* Fig. 5 of IS 1260 (Part 1)].

NOTES

1 Necessary safeguards against the risk arising from the storage and handling of large volumes of flammable liquids shall be provided and all due precautions shall be taken at all the times to prevent accidents by fire and explosion.

2 Except when they are opened for the purpose of cleaning and rendering them free from vapour, all empty tanks or other containers shall be kept securely closed unless they have been cleaned and free from vapour.

5.2.2 *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

5.2.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in Annex F of IS 229.

6.2 Criteria for Conformity

The lot shall be declared as conforming to the standard, if the test results of the composite test sample satisfy the requirements prescribed under **3.2**. Otherwise the lot shall be rejected.

7 QUALITY OF REAGENTS

7.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF RELATIVE DENSITY

A-1 OUTLINE OF THE METHOD

In this method mass of equal volumes of the material and water are compared at 27°C.

A-2 APPARATUS

A-2.1 Relative Density Bottle —25-ml capacity.

A-2.2 Water Bath —maintained at 27.0 ± 0.2°C.

A-2.3 Thermometer — any convenient thermometer of a suitable range with 0.1°C or 0.2°C subdivisions.

A-3 PROCEDURE

Clean and dry the relative density bottle. Weigh and fill with recently boiled and cooled water at 27°C. Fill to overflowing by holding the relative density bottle on its side in such a manner as to prevent entrapment of air bubbles. Insert the stopper and immerse in the water-bath. Keep the entire bulb covered with water and hold at that temperature for 30 min. Carefully remove any water, which has exuded from the capillary opening. Remove from the bath, wipe completely, dry and weigh. Again clean and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

A-4 CALCULATION

$$\text{Relative density at } 27°/27°\text{C} = \frac{M_1 - M_2}{M_3 - M_2}$$

where

M_1 = mass in g of the relative density bottle with the material,
M_2 = mass in g of the relative density bottle, and
M_3 = mass in g of the relative density bottle with water.

ANNEX B

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF DISTILLATION RANGE

B-1 PROCEDURE

Determine the distillation range by the procedure as prescribed in IS 5298 applying the following corrections.

B-1.1 Correction for Thermometer Reading

B-1.1.1 *Error of Scale*

In all the thermometer readings, make the corrections as indicated on the certificate of the thermometer.

B-1.2 Corrections for Barometric Pressure

If the barometric pressure prevailing during the determination is 760 mm Hg, no correction need be applied to the specified temperature and the thermometer scale as corrected for error of scale may be used as such. If however the prevailing barometric pressure deviates from 760 mm Hg, the specified temperature shall also be corrected as follows:

a) For every mm above 760 mm Hg, subtract 0.045°C from the specified temperature; and

b) For every mm below 760 mm Hg, add 0.045°C to the specified temperature.

NOTE —These corrections are valid only for pressure above 700 mm Hg.

ANNEX C

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF WATER CONTENT

C-1 GENERAL

Water is determined by the Karl Fischer method.

C-2 PROCEDURE

Take about 20 g of the material, weigh accurately and determine the water content by the procedure given in IS 2362.

ANNEX D

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF ACIDITY AS ACETIC ACID

D-1 OUTLINE OF THE METHOD

The material is titrated with standard sodium hydroxide solution to the phenolphthalein end point and from the volume of standard sodium hydroxide solution used, acidity is calculated as acetic acid.

D-2 REAGENTS

D-2.1 Sodium Hydroxide Solution — Approximately 0.01 N.

D-2.2 Hydrochloric Acid — Approximately 0.01 N.

D-2.3 Phenolphthalein Indicator — 0.5 percent (m/m) in isopropyl alcohol or rectified spirit.

D-2.4 Neutralized Isopropyl Alcohol — Add few drops of phenolphthalein indicator in 50 ml of isopropyl alcohol and add drop-wise 0.01 N sodium hydroxide solution till pink colour persists. Neutralized excess of sodium hydroxide by adding 0.01 N hydrochloric acid until the pink colour has just disappeared.

D-3 PROCEDURE

Weigh accurately about 100 g of the material to the nearest 0.1 g in a 500-ml conical flask. Add 50 ml of neutralized isopropyl alcohol and few drops of phenolphthalein indicator. Titrate with 0.01 N sodium hydroxide solution using a microburette, until the pink colour persists for 15 s.

D-4 CALCULATION

Acidity (as acetic acid), percent by mass

$$= \frac{6 \times V \times N}{M}$$

where

V = volume in ml of standard hydroxide solution,

N = normality of standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

ANNEX E

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF NON-VOLATILE MATTER

E-1 PROCEDURE

From a graduated measuring cylinder (*see* IS 878) introduce 100 ml of the sample into a 125 ml weighed to the constant mass platinum, silica or borosilicate glass evaporating dish, which has previously been heated to a temperature of 105 to 110°C and cooled in a desiccator. Evaporate the sample to dryness on a hot water bath and place the dish in an oven at a temperature of 100 ± 2°C. Cool the dish in a desiccator and weigh along with non-volatile matter to the nearest 0.1 mg. The difference in mass in g when multiplied by 1 000 gives mg/100 ml of the non-volatile matter.

ANNEX F

[*Table* 1, *Sl No.* (vii & viii)]

DETERMINATION OF PURITY AND METHYL iso-BUTYL CARBINOL

F-1 OUTLINE OF THE METHOD

A small volume of methyl iso-butyl ketone is injected into the gas chromatograph containing a capillary column with CP Wax 57 CB as stationary phase. Quantitative results are obtained by using suitable electronic integrator and internal normalization method which gives direct concentration of each impurity along with the chromatogram, area, retention time, etc.

F-2 APPARATUS

F-2.1 Gas Chromatograph — Any instrument having a hydrogen flame ionization detector that may be operated at the conditions given below:

Detector : Flame ionization
Column : Fused silica/WCOT
Length : 25 m
I.D. : 0.23 mm
Stationary phase : CP Wax 57 CB
Film thickness : 0.23 µm
Injector type : Splitter
Temperature :
  Injection port : 250°C
  Detector : 250°C
  Oven initial : 50°C
Initial time : 0 min
Programming rate : 0°C /min
Final temperature : 50°C
Final time : 0 min
Carrier gas : Nitrogen
Flow rate : Vent flow 150 ml/min
Make up gas/ Flow rate : 25 ml/min
FID hydrogen : 25 ml/min
Air : 250 ml/min
Sample size : 1.0 µl

NOTE—The above gas chromatographic conditions are suggestive, however the packed columns containing carbowax 20 M or OV 17 as stationary phases may be used provided standardization/calibrations are done after setting up chromatographic conditions for the required resolution.

F-3 REAGENTS

Nitrogen Chromatographic Grade
Chemicals for Calibration:
  Methyl iso-butyl ketone (MIBK)
  Methyl iso-butyl carbinol (MIBC)
  Mesityl oxide (MO)

F-4 CALIBRATION/ANALYSIS

Prepare a synthetic blend containing known quantities of MIBC, MO in MIBK. Set the chromatograph and the electronic integrator, as required and charge 1 microlitre of sample. Allow the chromatogram to develop. At the end of the run stop the integrator. Run the calibration blend in duplicate. Calculate the internal normalization factor by dividing the amount/area of MIBC, MO by the amount/area of MIBK. Find out the mean factor by averaging.

Charge the sample in the similar manner as given in the calibration above and find out the average area of MIBC, MO present in the sample.

F-5 CALCULATION

Percent m/m MIBK, MO (impurities) = Average area of EMP × Factor

Purity of MIBK = 100-impurities – Water

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 323 01 31, 323 33 75, 323 94 02

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices :		Telephone
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Eastern	: 1/14 C. I.T. Scheme VII M, V. I. P. Road, Kankurgachi CALCUTTA 700 054	337 84 99, 337 85 61 337 86 26, 337 91 20
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